Preparation and Properties of Flame-retardant Viscose Fiber Modified with Poly[bis(methoxyethoxy)phosphazene]

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Abstract: Poly[bis(methoxyethoxy)phosphazene] (PMEP) was synthesized and applied to the preparation of flame-retardant viscose fiber using wet spinning method. The structure of PMEP was characterized by FTIR and NMR. The combustibility and thermal stability of the flame-retardant viscose fiber containing different percentage of PMEP (0-20 %) were evaluated by limit oxygen index (LOI), 45° slope burning method, differential scanning calorimeter (DSC), and thermal gravity analysis (TGA). The results showed that the LOI of the flame-retardant viscose fiber containing 10 % or more PMEP could reach at least 28 % after normal family laundering, and the combustibility of the flame-retardant viscose fiber could reach the Japan industry standard JIS L1091-D (3 times of ignition). The analysis of the fiber thermal degradation suggested that PMEP as a macromolecular flame retardant had a multifunction of heat absorption, catalytic dehydration, carbonization, condensation-phase and gas-phase flame retardancy. The mechanical properties of the flame-retardant viscose fiber decreased insignificantly compared with that of virgin viscose fiber.

Keywords: Flame retardant, Viscose fiber, Polyphosphazene, Thermal properties, Degradation

Introduction

Viscose fiber is wildly used to manufacture textile clothing and decorative fabric due to its excellent hygroscopicity, breathability, and dyeability. Nowadays viscose fiber is still the most important alternative for cotton and other natural fibers in many countries. However, a disadvantage of viscose fiber is that the fiber can be easily ignited. From the consideration of personal safety, it is necessary to reduce the flammability of viscose. So the synthesis, processing methods and the main application properties of the flame retardants and flame-retardant viscose fiber had been reported extensively. For example, the flame-retardant viscose fabric finished with flame retardants, such as Proban and Pyrovatex CP [1,2], displays some disadvantages that the reaction conditions were rigorous and the mechanical properties sharply decreased, though the flame-retardant properties are improved. Therefore, the other researchers preferably used the wet spinning method to prepare flame-retardant viscose fiber. In this process, the viscose solution was blended with flame retardants [3].

The halogen flame retardant has excellent flame-retardant properties, but it tends to release toxic gases when it is burned, which can result in environmental pollution and personal injury [4]. Non-halogen flame retardants containing phosphorus, nitrogen or silicon are widely employed in flame-retardant process of textile materials as lower toxic additive [5-7]. Meanwhile, many nanocomposites are also developed as economical and environmentally friendly flame retardants, in recent years. However, the addition of nanocomposites can lead to obvious strength loss of the fiber [8].

Cyclotriphosphazene derivatives with small molecule weight have been used to process viscose fiber as effective flame retardants because of their high content of phosphorus-nitrogen elements and low toxicity [3,9]. Polyphosphazene derivatives also have been extensively studied and applied in many industrial areas such as catalysts [10], electrolytes [11], medical materials [12], etc. But there is barely reported about the preparation and the properties of flame-retardant viscose fiber modified with macromolecular polyphosphazene. The side chlorine atoms on the main chain of polydichlorophosphazene (PDCP) molecule can be substituted by different organic groups to obtain various polyphosphazene derivate. While the hydrophobicity and hydrophilicity of polyphosphazene derivate would change the compatibility between polyphosphazene derivate and viscose solution, which can result in decrease the quality of the fiber, such as difficult blending with viscose solution, loss of the polyphosphazene derivate during wet spinning, and uneven distribution of the polyphosphazene derivate throughout the viscose fiber.

In this paper, poly[bis(methoxyethoxy)phosphazene] (PMEP) was synthesized and applied to the preparation of flame-retardant viscose fiber via blending wet spinning method. The structure of PMEP and the modified fibers were characterized. The combustibility, thermal stability, mechanical property and durability of the flame-retardant viscose fiber were evaluated.

Experimental

Materials

Hexachlorocyclotriphosphazene (HCCP) was purchased from Hubei Yuancheng Pharmaceutical Co., Ltd. 2-methoxyethanol, sodium metal and THF were purchased from Kelong Chemical Reagent Company. Viscose solution

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(cellulose content 9 %), solidifying bath (H_2SO_4 130 g/l, Zn_2SO_4 10 g/l, Na_2SO_4 270 g/l) and RX-702 viscose fiber oiling agent were offered by Qingbai River Chemical Fiber Factory of Chengdu.

Synthesis of PMEP

The PMEP was synthesized according to the previous published method [13]. There crystallized HCCP was packed into an ampere bottle, filled with nitrogen gas after pumping vacuum and then flame sealed. The ampere bottle containing HCCP was placed into a muffle furnace to react under 240 °C for 5 h.

The sodium methoxyethoxide was prepared by reflux reaction of 2-methoxyethanol with sodium metal in THF at 50 °C for 5 h under nitrogen atmosphere.

The polydichlorophosphazene (PDCP) obtained was dissolved in THF and the THF solution of PDCP was put into a four-neckround-bottomed flask. The THF solution of sodium methoxyethoxide was added into the flask and reflux reacted at 68 °C for 10 h under nitrogen atmosphere. THF was vacuuming distillated after the end of reaction. The reacted product was settling out with heptane, neutralized to pH=7 by dilute hydrochloric acid neutralization. Water was added to dissolve the sediment, and then the solution was heated to separate the PMEP. The viscosity-average molecular weight of PMEP was 22127 dl/g.
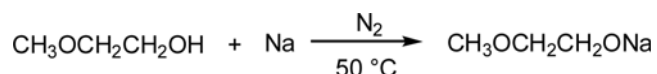
The synthesis procedure of PMEP was as following schemes.

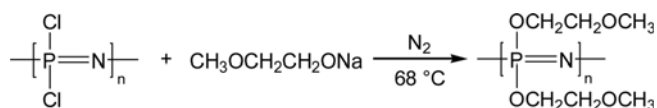**Preparation of the Flame-retardant Viscose Fiber**

Different ratios (0, 5, 10, 15, and 20 %) of PMEP were added to the solution and stirred for 30 min. After filtrated, defoamed and maturated, the mixed solution was added into
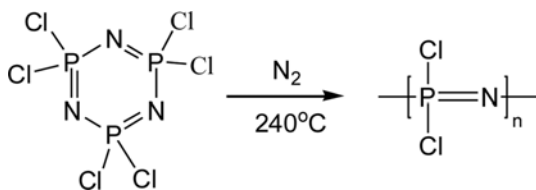
Scheme 1. Synthesis process of PDCP.

Scheme 2. Synthesis process of sodium methoxyethoxide.

Scheme 3. Synthesis process of PMEP.

the vessel containing viscose spinning solution. And the mixed spinning solution was spun through a 0.08 mm×200 holes spinneret. The fiber obtained with a spinneret draw ratio 0.92:1 and plasticizing draw ratio 1.3:1, was treated by normal treatment of desulphurizing, washing, oiling, etc.

Measurements

^{31}P-NMR spectra were recorded on Avance II-400 MHz spectrometers (Bruker Company, Switzerland). Fourier transform infrared spectra (FTIR) were recorded on a Nexus670 spectrometer (Nicolet Company, America). Elemental analyses were determined with an Euro EA 3000 elemental analyzer (Leeman Labs Inc., America). Scanning electron microscopy (SEM) images were obtained on a JSM-5900LV scanning electron microscopy (SEM) (JEOL Ltd., Japan).

Limiting oxygen index (LOI) of the fiber samples was tested with the HC-2C oxygen index instrument (Nanjing Shangyuan Analytical Instruments Company of China). The 45° slope burning method (Japan Industry Standard JIS L1091-D) [14] was adopted to evaluate the combustibility of the fiber samples.

The mechanical properties were tested with YG-001A Fiber Electronic Tension Meter (Taicang Textile Instrument Company, China). The weight of the 100 fiber samples with a length of 2 cm was tested with JN-B Precision Torsion Type Balance (Shanghai Precision & Scientific Instrument Company, China), and the linear density was calculated. The moisture regain was tested under standard conditions with the DHS20-1 Multi-function Intra Moisture Content Tester (Shanghai Precision & Scientific Instrument Company, China). Sound velocity values of fiber samples were measured 5 times with SCY-III sound velocity meter (Materials College Donghua University, China), and the average value was calculated as the orientation degree of fibers.

Differential scanning calorimeter (DSC) was measured under nitrogen atmosphere with DSC-60 thermal analyzer (Shimadzu International Trading Company, Japan). The measurement was carried with a temperature range from ambient temperature to 500 °C, heating rate 10 °C/min, and sampling temperature range from 50 °C to 500 °C. Thermal Gravity Analysis (TGA) was measured under nitrogen atmosphere with a DTA-60 thermal analyzer (Shimadzu International Trading Company, Japan). The test temperature range was from ambient temperature to 500 °C, the heating rate was 10 °C/min, and sampling temperature range was from 100 °C to 500 °C.

Results and Discussion

Characterization of PMEP

As shown in Figure 1, the FTIR spectrum of PDCP, it could be found that the absorption peaks of P=N, P-N and P-Cl appeared at 1231 cm^{-1}, 760 cm^{-1}, and 581 cm^{-1}, 522 cm^{-1} respectively [15]. The characteristic absorption peak located

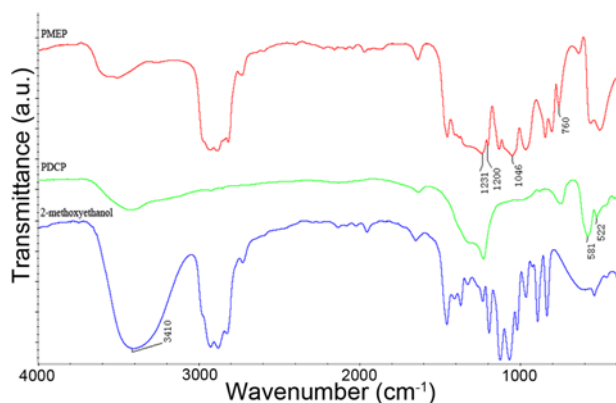at 3410 cm[-1] on the FTIR spectrum of 2-methoxyethanol was assigned to the -OH of 2-methoxyethanol. While from

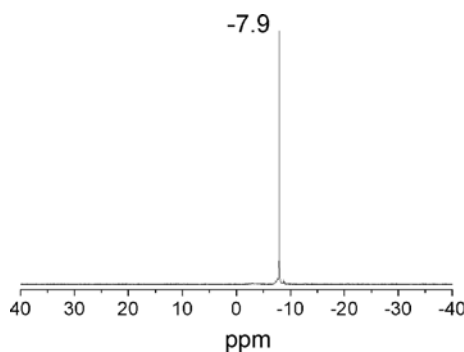**Figure 1.** FTIR spectrum of PDCP, 2-methoxyethanol and PMEP.

Figure 2. ^{31}P-NMR spectra of PMEP.

the FTIR spectrum of PMEP, it could be found that the absorption peaks of P-Cl at 581 cm[-1] and 522 cm[-1] disappeared completely [16], the characteristic peak of -OH at 3410 cm[-1] became weak and two new absorption peaks attributed to the P-O-C appeared at 1200 cm[-1] and 1046 cm[-1], which suggested that the chlorine atoms in PDCP had been substituted by methoxyethoxy groups. In other words, the bond of P-Cl was changed to the new chemical bonds of P-O-C.

According to reference 17, the ^{31}P-NMR peak of PDCP should be located at -19 ppm. After the chlorine atoms in PDCP were substituted by methoxyethoxy groups, the ^{31}P-NMR peak of PDCP disappeared and a new resonance peak assigned to PMEP appeared at -7.9 ppm [15] as shown in Figure 2. The reason may be that the change of electron density and conjugation of PDCP caused its ^{31}P-NMR peak taking place a chemical shift from -19 ppm to -7.9 ppm, after the electronegative substituents of PDCP replaced by methoxyethoxy groups.

The elemental analysis was shown in Table 1. The measured value was similar to the theoretical value of element content in PMEP, which confirmed the structure of PMEP.

Microstructure of the Viscose Fiber

The surface images of viscose fiber containing 0, 5, 10, 15 and 20 % PMEP were shown as (a), (b), (c), (d) and (e) in

Table 1. Elemental data of PMEP

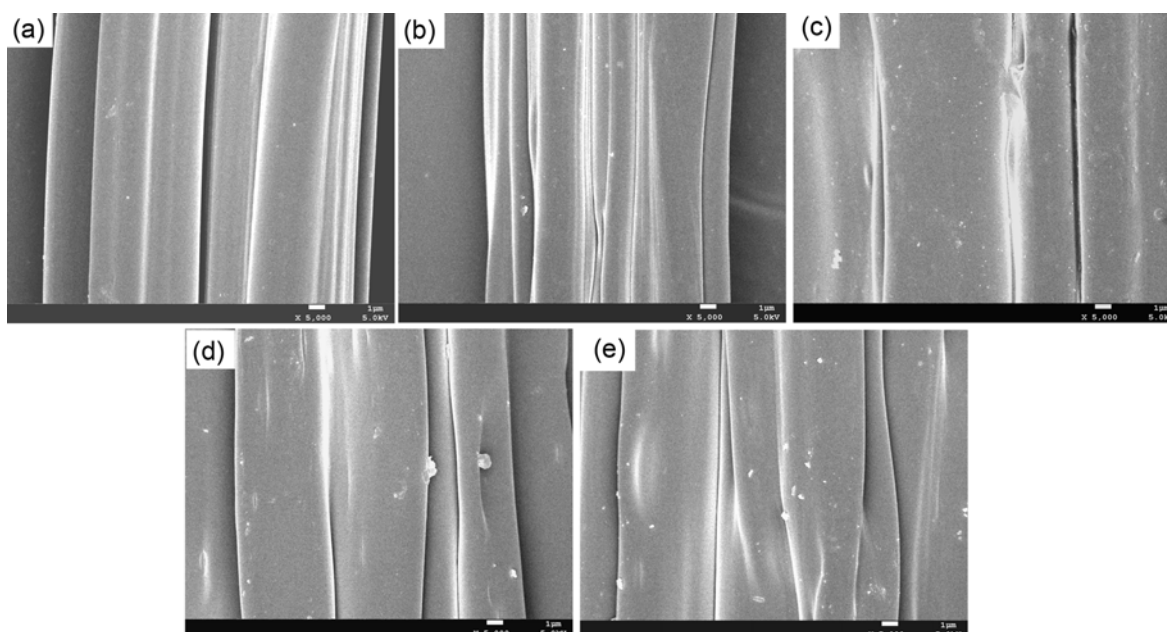| Element	C (%)	H (%)	N (%)
Theoretical value	36.92	7.18	7.18
Measured value	36.61	7.12	6.95

Figure 3. SEM of the viscose fiber modified with different contents of flame retardants; (a) 0 %, (b) 5 %, (c) 10 %, (d) 15 %, and (e) 20 %.

Figure 3, respectively. From the SEM images, it could be intuitively found that the virgin viscose fiber (0 % PMEP) was smooth, tidy and uniform. However, the surface of viscose fiber modified with flame retardants became rough and uneven, which had some obvious grooves and cracks. The reasonable explanation was that the temperature of solidifying bath was higher than that of PMEP phase transition temperature, the PMEP in the fiber taken place a phase separation in solidifying bath and could not completely co-oriented with the axial of cellulose macromolecules during drawing, which resulted in the rough of the fiber and grooves occurring. The addition of PMEP could decrease the concentration of cellulose sulfonic acid ester in the spinning solution, which effectively contributed to the fast shrinkage of fiber in the solidifying bath, and made the fiber surface to become more unevenness and irregular.

Combustibility of the Flame-retardant Viscose Fiber

The combustibility of the viscose fiber with different contents of PMEP was shown in Table 2. The data in Table 2 showed that the LOI values of the viscose fiber gradually increased with the increase of the PMEP contents. When the content of PMEP was over 5 %, the LOI value of the viscose fiber had reached 28 %, which was close to the content of oxygen in the air, and achieve the general requirement of flame-retardant standard. From the combustion phenomenon, it could be found that the controlled samples of viscose fiber smoldered slowly with obvious smoking. While the viscose fiber containing PMEP could rapidly char without smoking. When the viscose fiber contained more than 10 % PMEP, the times of ignition of the viscose fiber could achieve the Japan industry standard JIS L1091-D. According to the standard, the fiber with more than 3 ignition times could be regarded as the flame-retardant fiber. However, when the content of PMEP was below 10 %, the times of ignition of the viscose fiber were lower than 2 times. The results indicated that PMEP could improve the LOI value and char yield of the viscose fiber, therefore the viscose fiber modified with PMEP exhibited excellent flame retardancy.

From the data in Table 2, it also could be found that, before the samples were strictly washed according to the method of BS 5651-1978 [18] which was the equivalent of 50 times normal family laundering, the LOI value of the viscose fiber containing 0, 5, 10, 15, and 20 % PMEP was 19, 27, 31, 33, and 35 % respectively. And the same parameters of washed

samples became to 19, 26, 29, 31, and 32 % respectively. The decrease of the LOI values was only 3 % or less. The LOI value of flame-retardant viscose fiber containing PMEP over 5 % still maintained more than 28 % after washing, which indicated the flame-retardant viscose fiber had durable flame retardancy. Considering the performance and cost of the flame-retardant viscose fiber, 15 % was a proper percentage for the preparation of flame-retardant viscose fiber.

Thermal Stability of the Flame-retardant Viscose Fiber

The DSC curves of viscose fiber samples in nitrogen atmosphere were shown in Figure 4. The data of peak temperatures and enthalpies were shown in Table 3. The sample containing 0 % flame retardants was virgin viscose fiber. The sample containing 100 % flame retardants was the pure PMEP. It could be found that there was a strong and wide exothermic peak at 373.52 $^{\circ}$C on the DSC curve of virgin viscose fiber. At this temperature, the cellulose macromolecule began to decompose into laevoglucose, monosaccharide derivatives, and oligosaccharide mixture, and then laevoglucose, the main product decomposed into primary char and smaller molecules product such as CO. The exothermic peak located at the 498.61 $^{\circ}$C on the DSC curve of virgin viscose fiber suggested that these products were further oxidized into secondary char or CO_2 at higher temperature. And it was different from the original sample
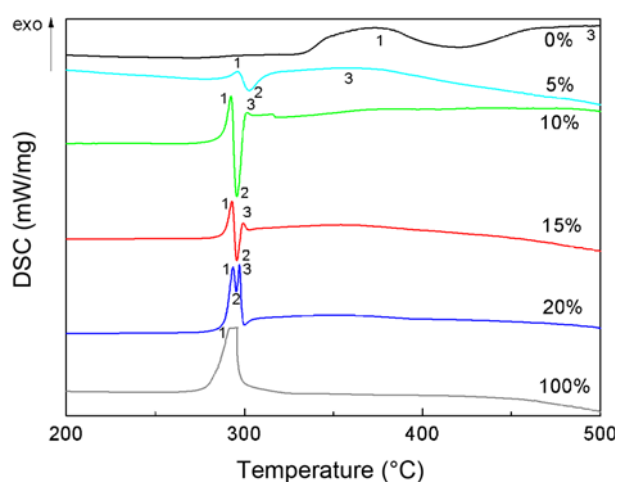

Figure 4. DSC curves of the viscose fiber containing different contents of flame retardants.

Table 2. Combustibility properties of viscose fiber samples

Contents of PMEP (%)	LOI (%, unwashed)	LOI (%, washed)	Times of ignition	Limit combustion phenomenon
0	19	19	1	Smoldering slowly with smoking
5	27	26	2	Charring rapidly with small smoking
10	31	29	3	Charring rapidly with no smoking
15	33	31	5	Charring rapidly with no smoking
20	35	32	8	Charring rapidly with no smoking

Table 3. Exothermic peak temperatures and enthalpies of viscose fiber samples

Contents of PMEP (%)	Main peak temperatures (^{o}C)			Enthalpies (J/g)		
	1	2	3	1	2	3
0	373.52	-	498.61	120.29	-	130.93
5	296.02	302.81	358.74	17.45	-21.82	40.03
10	292.54	295.76	301.47	61.40	-35.52	0.32
15	293.06	295.77	299.23	45.85	-21.89	12.76
20	293.70	295.36	297.14	10.75	-4.77	6.36
100	296.30	-	-	707.15	-	-

that, on the DSC curves of the viscose fiber samples containing 5-20 % PMEP, the exothermic peaks appeared at 296.02 ^{o}C, 292.54 ^{o}C, 293.06 ^{o}C, 293.70 ^{o}C, respectively. While the endothermic peak located at 302.81 ^{o}C, 295.76 ^{o}C, 295.77 ^{o}C, 295.36 ^{o}C, respectively. The result showed that the addition of PMEP could make the decomposition temperature of viscose fiber to move up about 70 ^{o}C. Compared with the exothermic peak at 296.30 ^{o}C of the pure PMEP, the first exothermic peak of the viscose fiber samples modified with PMEP indicated that the exothermic pyrolysis reaction of PMEP began from the temperature. The endothermic chemical reaction between the pyrolysis products of PMEP and cellulose occurred at the temperature about 295 ^{o}C. These reactions could result in the decomposition temperature of cellulose which degraded to laevoglucose decreasing from the 373.52 ^{o}C of the virgin viscose fiber to 300 ^{o}C. The temperature change of exothermic peaks indicated that PMEP promoted the cellulose to decompose under a low temperature. The degradation products of PMEP could be the materials of phosphoric acid, metaphosphoric acid, and polymetaphosphoric acid, which had strong dehydration function. Therefore PMEP with function of dehydrating and charring could reduce the production of CO and promote cellulose charring. The charring products from cellulose and the polymetaphosphoric acid from the decomposition of PMEP could effectively isolate oxygen and avoided continuous combustion, which was provided with the condensation-phase flame-retardant effect. On the other hand, PMEP broke down in the flame to small molecular species such as P_2, PO, PO_2 and HPO_2. These species cause the hydrogen atom concentration in the flame to be reduced and inhibited the reaction of a hydrogen atom with an oxygen molecule to give a hydroxyl radical thus quenching the flame [19]. The N_2, NH_3, and NO_2 released from the burning of PMEP, as inert gases which were hard to burn, played the roles of diluting oxygen. It is the gas-phase flame-retardant effect.

From the data as shown in Table 3 and Figure 4, compared with that of virgin viscose fiber, the enthalpies of corresponding exothermic peak1 of viscose fiber containing PMEP were lower which exhibited that PMEP could promote the dehydration of cellulose and reduce heat release of viscose fiber. The enthalpies corresponding exothermic peak 3 of
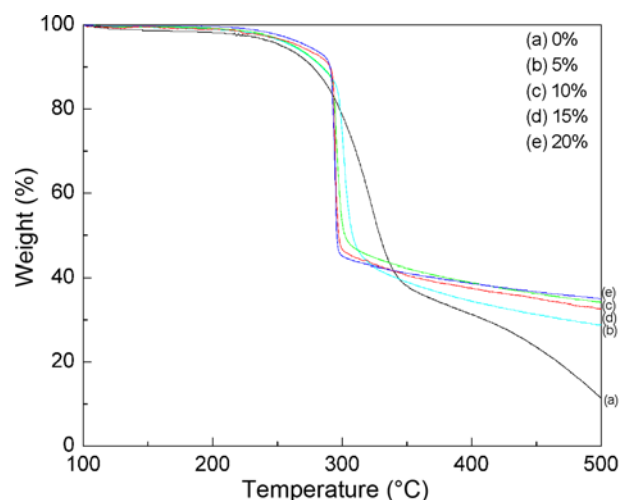
Figure 5. TGA curves of the viscose fiber containing different contents of flame retardants.

modified viscose fiber were also lower than that of virgin viscose fiber, it indicated the further decomposition of modified samples at higher temperature became weak, relative to that of virgin viscose fiber. Therefore the addition of PMEP could convert the combustion mechanism of cellulose fiber.

TGA curves of viscose fiber containing different contents of PMEP were as shown as in Figure 5 and the relative characteristic parameters as shown in Table 4. The main weight loss stage (100-338 ^{o}C) of the viscose fiber samples indicated the primary decomposition and charring of cellulose. At this stage, the temperature of maximum weight loss rate occurred at 324.51 ^{o}C and the maximum weight loss rate was 11.08 %/min for virgin viscose fiber. At this stage, the termination temperature of decomposition and the weight loss of virgin viscose fiber was 338.33 ^{o}C and 61.177 %, respectively. The temperature of maximum weight loss rate for viscose fiber containing PMEP (5-20 %) was 301.16, 295.87, 294.78, 294.19 ^{o}C, respectively, and the maximum weight loss rate of modified samples was 37.27, 60.13, 101.10, 153.70 %/min, respectively. Compared with that of virgin viscose fiber, it could be found that the temperature of maximum weight loss rate of viscose fiber modified with PMEP was lower 24-30 ^{o}C than that of virgin viscose fiber,

while the maximum weight loss rate were higher 26-142 %/min than that of virgin viscose fiber. The temperature of maximum weight loss rate deceased and weight loss rate increased with the increase of PMEP content for the modified samples. At this stage, the termination decomposition temperature and the weight loss percentage of modified viscose fiber were lower than that of virgin viscose fiber. These results indicated that the addition of PMEP could reduce the temperature of maximum weight loss rate and the termination decomposition temperature of viscose fiber, promote the cellulose to dehydrate into laevoglucose, and further form the char at the relative temperature.

From Figure 5 and the relative data in Table 4, it could be found that the further decomposition and charring of cellulose occurred at the end stage (295-500 °C). Contrary to that of viscose fiber modified with PMEP, the decomposition rate of virgin viscose fiber was relatively rapid and its weight loss percentage was higher than that of the modified samples. While the remained mass of controlled sample was lower than that of the modified samples. This phenomenon attributed to the small flammable molecular product derived from further decomposition of virgin viscose fiber, which could result in cellulose fiber continuing combustion and further weight loss.

Mechanical Properties

The data in Table 5 showed the physical-mechanical properties of viscose fiber modified with PMEP. It could be found that with the increase of the PMEP content, linear density of viscose fiber increased, while the moisture regain, breaking strength, breaking elongation and orientation degree of the viscose fiber decreased slightly. It might be that the

addition of PMEP would lead to the weight of unit length and caused the linear density of the viscose fiber increasing. On the other hand, the addition of PMEP would obstruct the stretch orientation of cellulose macromolecules and cause some interior defects for viscose fiber during drawing, which resulted in the decrease of breaking strength and breaking elongation. But the mechanical properties of viscose fiber modified with PMEP decreased insignificantly, and the modified viscose fiber still was at an acceptable level for needs of fire fighting and military protection.

Conclusion

Flame-retardant viscose fibers containing different contents of PMEP were prepared via blending wet spinning method. The LOI value of viscose fiber containing only 10 % PMEP could reach 28 % and that of the sample containing 20 % PMEP was as high as 35 %. The 15 % PMEP addition was proper for the preparation of flame-retardant viscose fiber. The viscose fibers containing more than 10 % PMEP could survive more than 3 times of ignition and the LOI value could maintain over 28 % after 50 times wash tests, which suggested the viscose fiber modified by this method had durable flame retardancy.

The results of the DSC and TGA indicated that the PMEP could begin chemical reaction at nearby 295 °C, and the addition of PMEP could promote cellulose decomposing and dehydrating during the fiber burning or heating process. The decomposing and dehydrating temperatures of the viscose fiber modified with PMEP was lower about 70 °C than that of the virgin viscose fiber. The weight loss percentage of viscose fiber modified with PMEP was less 12 % than that

Table 4. Characteristic data of TGA graphs of viscose fiber samples

Contents of PMEP (%)	Main stage				End stage	Remained mass (%)
	Temperature of maximum weight loss (%)	Maximum weight loss rate (%/min)	Decomposition ended temperature (°C)	Weight loss percentage (%)	Weight loss percentage (%)	
0	324.51	11.08	338.32	61.177	27.477	11.346
5	301.16	37.27	307.52	53.693	17.522	28.785
10	295.87	60.13	300.54	51.531	14.317	34.152
15	294.78	101.10	296.57	53.749	13.669	32.582
20	294.19	153.70	294.90	54.423	10.556	35.021

Table 5. Physical-mechanical properties of viscose fiber samples

Contents of PMEP (%)	Linear density (dtex)	Moisture regain (%)	Breaking strength (cN/dtex)	Breaking elongation (%)	Degree of orientation
0	5.80	14.4	1.35	33.07	0.510
5	5.90	14.2	1.29	27.40	0.497
10	5.95	14.1	1.26	24.97	0.421
15	6.03	13.8	1.17	22.63	0.407
20	6.18	13.8	1.12	22.36	0.386

of the virgin viscose fiber, while the remained mass of the viscose fiber modified with PMEP was more 16 % than that of the virgin viscose fiber.

In spite of the mechanical properties decreasing slightly compared with that of virgin viscose fiber, the results suggested that PMEP as a macromolecular flame retardant had good combination with viscose fiber and exhibited good durable flame retardancy.

Acknowledgment

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